

PATENT ABSTRACTS OF JAPAN

(11)Publication number : 06-227886

(43)Date of publication of application : 16.08.1994

(51)Int.Cl.

C30B 9/04
 C30B 11/14
 C30B 17/00
 C30B 19/12
 C30B 23/02
 C30B 29/36
 // H01L 21/203
 H01L 21/208

(21)Application number : 05-018272

(71)Applicant : TOSHIBA CORP

(22)Date of filing : 05.02.1993

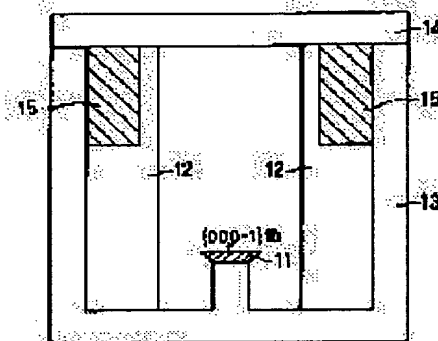
(72)Inventor : UEMOTO TSUTOMU

(54) PRODUCTION OF SEMICONDUCTOR SINGLE CRYSTAL

(57)Abstract:

PURPOSE: To provide the subject production method intended to suppress developing crystal defects, so designed that, for the side faces in contact with the main growth surface of a seed crystal, such seed crystal is provided so as to be slant from either plane, (0001)-plane or (1-100)-plane.

CONSTITUTION: Inside a crucible 13 set up in a single crystal production unit by sublimation method, shelves 12 made of porous graphite carbon are arranged, material powder 15 such as of SiC is put thereon, a seed crystal is placed, and the whole system is sealed by a crucible cover 14. For the seed crystal 11, (0001)plane is set as the main growth surface, the seed crystal is finished in a conical form with the side face, (1-100)-plane, slant by at least 3° from 90° such as 45° or 60° with the main growth surface, and placed at a specified location. Then, the material powder 15 is heated to about 2500° C, the seed crystal 11 to about 2300° C, to sublimate the material and grow the seed crystal 11, thus affording the aimed SiC single crystal with few crystal defects and the main growth surface retained flat.



LEGAL STATUS

[Date of request for examination] 30.08.1999

[Date of sending the examiner's decision of rejection]

[Kind of final disposal of application other than the examiner's decision of rejection or application converted registration]

[Date of final disposal for application]

[Patent number] 3251687

[Date of registration] 16.11.2001

[Number of appeal against examiner's decision of rejection]

[Date of requesting appeal against examiner's decision of rejection]

[Date of extinction of right]

Copyright (C); 1998,2003 Japan Patent Office

* NOTICES *

JPO and NCIPi are not responsible for any damages caused by the use of this translation.

- 1.This document has been translated by computer. So the translation may not reflect the original precisely.
- 2.**** shows the word which can not be translated.
- 3.In the drawings, any words are not translated.

CLAIMS

[Claim(s)]

[Claim 1] the side face which touches with a bulk grown method at the growth principal plane of said seed crystal in the manufacture approach of a semi-conductor single crystal of growing up a hexa GONARU mold single crystal, on the seed crystal which consists of a hexa GONARU mold single crystal — a field (0001) — and (1-100) The manufacture approach of the semi-conductor single crystal characterized by using the seed crystal which has only the field to which it inclined from any field of a field.

[Translation done.]

* NOTICES *

JPO and NCIP are not responsible for any damages caused by the use of this translation.

- 1.This document has been translated by computer. So the translation may not reflect the original precisely.
- 2.**** shows the word which can not be translated.
- 3.In the drawings, any words are not translated.

DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Industrial Application] This invention relates to the manufacture approach of the hexa GONARU mold semi-conductor single crystal manufactured by the sublimating method or the solution method.

[0002]

[Description of the Prior Art] Research is done as an environmental-proof component ingredient which can bear SiC also under elevated-temperature high pressure also thermally since it is chemical very stable. Moreover, on the other hand, as for SiC, for a certain reason, the energy gap attracts 2.3eV or more of attentions as a short wavelength light emitting device ingredient. Some crystal structures, such as a hexa GONARU mold (six law crystal system), and a cubic mold (cubic system), a ROMBOHEDORARU mold (trigonal system), exist in SiC. Since the energy gap of especially the single crystal of 6H mold (hexa GONARU mold which makes six molecules one period), or 4H mold (hexa GONARU mold which makes four molecules one period) is about 3eV in it, it is expected as an ingredient of blue LED.

[0003] and manufacture of blue LED — journal OBU Applied ones FIJIKUSU it is reported to 50(1979) pp 8215–8225 [Journal of Applied Physics 50(1979) pp 8215< 8225] — as — a liquid-phase-epitaxial method (LPE law) or Japanese journal OBU Applied one FIJIKUSU It is carried out by the chemical reaction depositing method (CVD method) as reported to 19(1980) ppL353 – L856[Japanese Journal of Applied Physics 19(1980) L353<L856]. In any case, LED is manufactured on the SiC single crystal substrate (0001) side of 6H mold.

[0004] Thus, the hexa GONARU mold SiC single crystal substrate has played the role important as a growth substrate of blue LED. The sublimating method which is made to sublimate SiC raw material powder as the growth approach of a hexa GONARU mold SiC single crystal substrate conventionally, and is deposited in a low temperature side is used. For example, it is stated to applied FIJIKUSU letter 58(1991) pp 56–58 [AppliedPhysics Letter 58(1991) pp 56< 58]. Invention-in-this-application persons created the large crystal of aperture with crystal growth die length using the diameter of a crystal becoming large, in order to enlarge the diameter of a crystal by the sublimating method. However, it is difficult for the breadth of a path to become small and to obtain the substrate of the diameter of macrostomia as crystal length becomes long by this approach. Moreover, the growth side became a concave and the trouble that the substrate of high quality could not be obtained was found out as crystal length became long.

[0005]

[Problem(s) to be Solved by the Invention] As mentioned above, when growing up the SiC single crystal of a hexa GONARU mold single crystal by the sublimating method, in order to obtain the substrate of the diameter of macrostomia, growth had to be lengthened enough. However, when growth die length was lengthened in this case, the crystal growth side turned into a concave surface, and had the problem that increase of the path of ***** stopped soon. Moreover, the crystal obtained had the problem that many crystal defects could not trust it in respect of quality. Moreover, the above-mentioned technical problem exists in various hexa GONARU mold semi-conductor single crystals other than SiC.

[0006] It was made in view of the above-mentioned trouble, the growth side of the crystal face prevents becoming a concave surface, and, as for this invention, a crystal defect aims the manufacture approach of the hexa GONARU mold semi-conductor single crystal of few diameters of macrostomia at offer.

[0007]

[Means for Solving the Problem] the side face in which the manufacture approach of the semi-conductor single crystal of this invention touches with a bulk grown method at the growth principal plane of said seed crystal in the manufacture approach of a semi-conductor single crystal of growing up a hexa GONARU mold single crystal, on the seed crystal which consists of a hexa GONARU mold single crystal in order to solve the above-mentioned trouble — a field (0001) — and (1–100) It is characterized by to use the seed crystal which has only

the field to which it inclined from any field of a field.

[0008]

[Function] As a result of research of this invention persons, compared with the crystal of the others in alpha-SiC for example, which is a hexa GONARU mold crystal, the most important point as a cause of concave-surface-izing of a growth side had the strong field bearing dependency of the rate of crystal growth, and found out that the flat surface called a facet appeared. It is a growth principal plane (1-100) about a field (0001) to drawing 11. The facet 111 at the time of making a field into the side face is shown. this invention persons — this phenomenon — $\langle 0001 \rangle$ or $\langle 1-100 \rangle$ since the growth rate of bearing is very slow — the field (0001) of the side face of a growth principal plane — or (1-100) I was going to extend in parallel with a field and thought that a facet appeared as a result. As for the single crystal with which, as for the part into which this facet 111 appears, a growth rate grows early rather than a growth principal plane, that periphery rises rather than a center section (112 is climax of a periphery). The growth principal plane of the single crystal obtained as a result found out becoming the concave in which the center section became depressed.

[0009] the side face in which artificers touch the growth principal plane of seed crystal in order to prevent the appearance of the above-mentioned facet — a field (0001) — and (1-100) It found out that it could attain by leaning from any field of a field.

[0010]

[Example] The example of this invention is explained below at a detail.

[0011] The 1st example of this invention is explained. Drawing 1 is the schematic diagram of the sublimating method single crystal manufacturing installation which is one of the bulk grown methods. For seed crystal and 12, as for crucible and 14, porous graphite and 13 are [11 / a crucible lid and 15] raw material powder. In this example, the hexa GONARU mold SiC single crystal was grown up.

[0012] The crystal mold of the hexa GONARU mold SiC seed crystal 11 and the relation of a growth principal plane are shown in drawing 2. Moreover, drawing which expresses the configuration of the SiC seed crystal 11 to drawing 3 is shown. The growth principal plane of seed crystal 11 is {000-1}. It considered as the field (0001) (carbon side), and operated orthopedically in a circle with a diameter of 10mm. Moreover, all side faces are {000-1}. It was processed to the field the appearance which becomes 45 degrees, and in the shape of [by which the head was crushed] a cone. The raw material was made to sublime on seed crystal 11 using the equipment of drawing 1 by having made the large field of this seed crystal 11 into the growth side, and the SiC single crystal was grown up. It was referred to as the temperature of 2300 degrees C of seed crystal, the raw material temperature of 2500 degrees C, and growth pressure 50torr as growth conditions. The SiC single crystal with a die length of about 20mm was grown up in growth time amount 20 hours.

[0013] It is a growth principal plane as the SiC single crystal 41 grown up into drawing 4 by this invention, and an example of a comparison {000-1} It considers as a field (0001) (carbon side), and is a part of side face (1-100). The sectional view of the SiC single crystal 43 grown up on the same conditions as the 1st example using the seed crystal 42 of the shape of a cylinder out of which the field has come is shown.

[0014] The SiC single crystal 43 which was grown up in the case of the example of a comparison (drawing 4 (b)) became a slack type, and that a path spreads soon has stopped. Moreover, the growth side became the concave which became depressed about 3mm in the center section, and abnormalities in a crystal, such as transition and twin crystal, have arisen with the stress by abnormality growth of the periphery section. On the other hand, in this invention (drawing 4 (a)), the SiC single crystal 41 grows in the shape of a cone. For this reason, the direction of this invention becomes large and the diameter of a crystal can enable manufacture of the diameter substrate of macrostomia. Moreover, it came out for concave surface-ization of a growth principal plane to be lost mostly, to become possible to suppress the increment in the defect by abnormality growth of the periphery section, and to improve the quality of a crystal.

[0015] The include angle of the side face at the time of making a growth principal plane into a field (0001) as seed crystal of this invention at drawing 5 is shown. Side face (1-100) It is desirable to lean at least 3 degrees or more to a field (0001) (for it to be 90 degrees to a field) (field in [A] drawing). It becomes possible by using such seed crystal to be able to suppress generating of a facet and to prevent concave surface-ization of a growth principal plane. Therefore, a hexa GONARU mold single crystal with few crystal defects can be grown up with the diameter of macrostomia. Next, the 2nd example of this invention is shown. At this example, it is a growth principal plane (11-20). The SiC single crystal was grown up using the SiC seed crystal made into the field. The equipment to be used performed growth conditions like the 1st example using what is shown in drawing 1.

[0016] It is the alpha-6HSiC crystal mold and growth principal plane of seed crystal of this example at drawing 6 (11-20). Drawing of the relation of a field is shown and it is a growth principal plane (11-20) to drawing 7. The include angle of the side face at the time of considering as a field is shown. Growth principal plane (11-20) When

it considers as a field, it is on a side face (1-100). It is 30 degrees, 90 degrees, and 150 degrees from a principal plane that a field and (0001) a field appear, and it should just lean 3 degrees or more from these fields, respectively (field in [A] drawing).

[0017] In this example, the growth principal plane was formed with a diameter of 10mm in the shape of a circle, and the SiC single crystal with a growth die length of 20mm was grown up by making the larger one of a field into a growth principal plane using the conic seed crystal with which the head which leaned 60 degrees of side faces from the growth principal plane was crushed. Also in this example, the SiC single crystal was able to be grown up in the shape of a cone as well as the 1st example. Moreover, the facet of the periphery section did not appear and concave surface-ization of a growth side did not take place. Next, the 3rd example of this invention is shown. At this example, it is a growth principal plane (1-100). The SiC single crystal was grown up using the SiC seed crystal made into the field. The equipment to be used performed growth conditions like the 1st example using what is shown in drawing 1.

[0018] It is the alpha-6HSiC crystal mold and growth principal plane of seed crystal of this example at drawing 8 (1-100). Drawing of the relation of a field is shown and it is a growth principal plane (1-100) to drawing 9. The include angle of the side face at the time of considering as a field is shown. Growth principal plane (1-100) When it considers as a field, it is on a side face (1-100). It is 60 degrees, 90 degrees, and 120 degrees from a principal plane that a field and (0001) a field appear, and it should just lean 3 degrees or more from these fields, respectively (field in [A] drawing).

[0019] In this example, the growth principal plane was formed with a diameter of 10mm in the shape of a circle, and the SiC single crystal with a growth die length of 20mm was grown up by making the larger one of a field into a growth principal plane using the conic seed crystal with which the head which leaned 45 degrees of side faces from the growth principal plane was crushed. Also in this example, the SiC single crystal was able to be grown up in the shape of a cone as well as the 1st example. Moreover, the facet of the periphery section did not appear and concave surface-ization of a growth side did not take place.

[0020] Next, the 4th example of this invention is shown. In this example, the principal plane of the SiC seed crystal used in the 1st, 2, and 3 above-mentioned example was ground further, and the SiC single crystal was grown up using the seed crystal which makes the field shifted 5 degrees from each principal plane a growth principal plane. Growth equipment used the thing of drawing 1 and made growth conditions the same thing as the 1st example. In this example, since the growth rate of a growth principal plane and a side face hardly changes, the form of a crystal can obtain breadth and a good single crystal from seed crystal in a natural cone form.

[0021] Next, the 5th example of this invention is shown. In this example, the SiC single crystal was deposited and grown up on SiC seed crystal from the solution of Si using the solution method which is one of the bulk grown methods. Drawing 10 is growth equipment used by this example. 100H growth is performed collecting the Si solvents 102 into the crucible 101 which consists of C or SiC, and using temperature of 1800 degrees C and seed crystal 103 as 1700 degrees C for the maximum elevated-temperature section in a solvent. C of a crucible and Si of a solvent deposit on the SiC seed crystal 103, and a single crystal grows. In this case, seed crystal processed the growth principal plane like the 1st example in the shape of [by which a field (0001) and 45 degrees of side faces were leaned from the growth principal plane, and the head was crushed in them] a cone. The larger one of the field of this seed crystal was made into the growth principal plane, and the SiC single crystal around 10mm was grown up on the SiC seed crystal 103 on the above-mentioned growth conditions. It also sets to this example and is on a side face (1-100). Compared with the thing using the seed crystal out of which the field has come, expansion of the path of a crystal could be aimed at sharply, and concave surface-ization of a growth side was not seen.

[0022] Next, the 6th example of this invention is shown. In this example, the single crystal of hexa GONARU mold gallium nitride was grown up using the sublimating method. Since the not much big crystal as seed crystal is not obtained, current gallium nitride needs to use a minute crystal as a seed. In this example, the growth principal plane was made into the field (0001), and the seed crystal into which the side face was processed in the shape of [by which the head which inclined 45 degrees from the growth principal plane was crushed] a cone was used. This side face is all (1-100). It is the leaning field from the field. Growth equipment is almost the same as what was used in the example 1, and, for growth temperature, a raw material is N₂ in 1300 degrees C and 1200 degrees C of seed crystal. It carried out on condition that the number Torr of pressures of an ambient atmosphere. Also in this example, increase of the path of a growth single crystal was achieved sharply, concave surface-ization of a growth side was not seen but improvement in quality was achieved.

[0023] In addition, although the growth principal plane used circular seed crystal in each example mentioned above, a trigonum, a rectangular head, a polygon, especially an ellipse form, etc. are not limited. the side face which in the case of a trigonum, a rectangular head, and a polygon touches a growth principal plane in seed crystal even if pillar-shaped — a field (0001) — and (1-100) It can be made only the field to which it inclined

from any field of a field. Moreover, the temperature of a crystal and a temperature gradient can be used in each above-mentioned example, changing them freely.

[0024]

[Effect of the Invention] By using this invention, as mentioned above, it comes out that prevent the growth side of the crystal face turning into a concave surface, and a crystal defect manufactures the hexa GONARU mold semi-conductor single crystal substrate of few diameters of macrostomia. Moreover, it comes out to be able to suppress the increment in the defect by the abnormality growth which a facet collides with, and to improve the quality of a crystal.

[Translation done.]

*** NOTICES ***

JPPO and NCIPi are not responsible for any damages caused by the use of this translation.

- 1.This document has been translated by computer. So the translation may not reflect the original precisely.
- 2.**** shows the word which can not be translated.
- 3.In the drawings, any words are not translated.

DESCRIPTION OF DRAWINGS

[Brief Description of the Drawings]

[Drawing 1] The schematic diagram of the sublimating method single crystal manufacturing installation used in the 1st, 2, 3, 4, and 6 example of this invention

[Drawing 2] Drawing showing the field (0001) of a hexa GONARU mold crystal

[Drawing 3] Drawing showing the configuration of the seed crystal used for the 1st example of this invention

[Drawing 4] The comparison Fig. of the SiC single crystal and the conventional example which were grown up in the 1st example of this invention

[Drawing 5] Drawing showing the include angle of the side face of the seed crystal when making a growth principal plane into a field (0001)

[Drawing 6] Hexa GONARU mold crystal (11-20) Drawing showing a field

[Drawing 7] Growth principal plane (11-20) Drawing showing the include angle of the side face of the seed crystal when considering as a field

[Drawing 8] Hexa GONARU mold crystal (1-100) Drawing showing a field

[Drawing 9] Growth principal plane (1-100) Drawing showing the include angle of the side face of the seed crystal when considering as a field

[Drawing 10] The schematic diagram of a solution method single crystal manufacturing installation used in the 5th example of this invention

[Drawing 11] a growth principal plane — ** (0001) — the side face (1-100) Drawing showing the facet which appears in the periphery section when considering as a field

[Description of Notations]

11. Seed Crystal
12. Porous Graphite
13. Crucible
14. Crucible Lid
15. Raw Material Powder
101. Graphite Crucible
102. Si Solvent
103. Seed Crystal

[Translation done.]

#7175

(19)日本国特許庁(JP)

(12) 公開特許公報(A)

(11)特許出願公開番号

特開平6-227886

(43)公開日 平成6年(1994)8月16日

(51)Int.Cl.⁵

識別記号

庁内整理番号

FI

技術表示箇所

C30B 9/04

11/14

17/00

19/12

23/02

9040-4G

審査請求 未請求 請求項の数1 OL (全6頁) 最終頁に続く

(21)出願番号

特願平5-18272

(22)出願日

平成5年(1993)2月5日

(71)出願人 000003078

株式会社東芝

神奈川県川崎市幸区堀川町72番地

(72)発明者 上本 勉

神奈川県川崎市幸区小向東芝町1番地 株

式会社東芝研究開発センター内

(74)代理人 弁理士 則近 憲佑

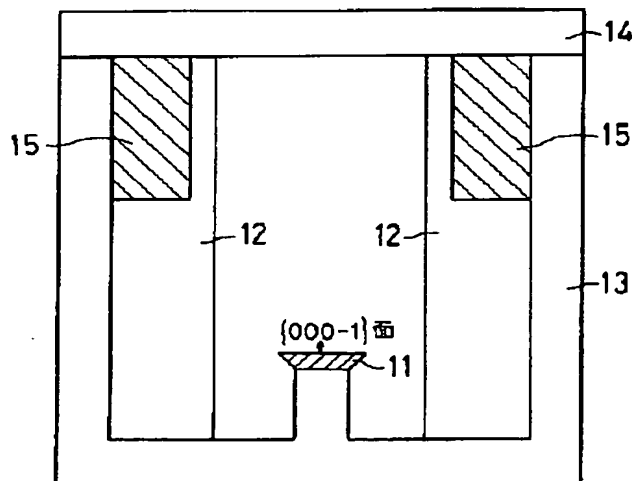
(54)【発明の名称】 半導体単結晶の製造方法

(57)【要約】

【目的】 本発明は、従来の単結晶より大口径で欠陥の少ないSiC単結晶基板を提供することを目的とする。

【構成】 SiCを昇華法または溶液法により成長する場合、種結晶の成長主面に接する側面を(0001)面及び(1-100)面のいずれの面からも傾いた面で形成する。

【効果】 ファセットによる結晶径の広がり阻害がないため、径の増大が容易であり、成長主面の平坦性が確保され、結晶欠陥の増加要因の1つがなくなる。



【特許請求の範囲】

【請求項1】 バルク成長法により、ヘキサゴナール型単結晶よりなる種結晶上にヘキサゴナール型単結晶を成長させる半導体単結晶の製造方法において、前記種結晶の成長主面に接する側面は(0001)面及び(1-100)面のいずれの面からも傾いた面のみを有する種結晶を用いることを特徴とする半導体単結晶の製造方法。

【発明の詳細な説明】

【0001】

【産業上の利用分野】 本発明は昇華法または溶液法により製造されたヘキサゴナール型半導体単結晶の製造方法に関する。

【0002】

【従来の技術】 SiCは熱的にも化学的にも非常に安定であるために、高温高压下でも耐え得る耐環境素子材料として研究がされている。また一方ではSiCはエネルギーギャップが2.3eV以上あるために短波長発光素子材料として注目を集めている。SiCにはヘキサゴナール型（六方晶系）やキュービック型（立方晶系）、ロンボヘドラル型（三方晶系）など幾つかの結晶構造が存在する。その中で特に6H型（6分子を1周期とするヘキサゴナール型）や4H型（4分子を1周期とするヘキサゴナール型）の単結晶はエネルギーギャップが約3eVであるため、青色LEDの材料として期待されている。

【0003】そして、青色LEDの製造はジャーナルオブ アプライド フィジクス 50(1979)pp8215~8225 [Journal of Applied Physics 50(1979)pp8215~8225] に報告されている様に液相エピタキシャル法（LPE法）もしくはジャパニーズ ジャーナル オブ アプライド フィジクス 19(1980)ppL353~L856 [Japanese Journal of Applied Physics 19(1980)L353~L856] に報告されている様に化学反応堆積法（CVD法）により行われており、いずれの場合も6H型のSiC単結晶基板(0001)面上にLEDが製造されている。

【0004】この様にヘキサゴナール型SiC単結晶基板は青色LEDの成長基板として重要な役割を果たしている。従来ヘキサゴナール型SiC単結晶基板の成長方法としてSiC原料粉末を昇華させて低温側に析出させる昇華法が用いられている。例えばアプライドフィジクスレター58(1991)pp56~58 [Applied Physics Letter 58(1991)pp56~58] に述べられている。本願発明者らは昇華法で結晶径を大きくするためには結晶成長長さとともに結晶径が大きくなることを利用して口径の広い結晶を作成した。しかしながらこの方法では結晶長が長くなるにつれて径の広がりが小さくなり大口径の基板を得ることが難しい。また結晶長が長くなるにつれて成長面が凹型になってしまい高品質の基板を得ることができないという問題点を見いだした。

【0005】

【発明が解決しようとする課題】 上述したように昇華法によりヘキサゴナール型単結晶のSiC単結晶を成長させる場合、大口径の基板を得るためには成長を十分長くしなければならなかった。しかしこの場合、成長長さを長くすると、結晶の成長面は凹面になり、やがては結晶の径の増大が止まるといった問題があった。また得られる結晶は結晶欠陥が多く品質面で信頼できないといった問題があった。また、上記した課題は、SiC以外の様々なヘキサゴナール型半導体単結晶に存在するものである。

【0006】本発明は上記問題点を鑑みなされたもので、結晶面の成長面が凹面になることを防ぎ結晶欠陥が少ない大口径のヘキサゴナール型半導体単結晶の製造方法を提供を目的とする。

【0007】

【課題を解決するための手段】 上記問題点を解決するために、本発明の半導体単結晶の製造方法は、バルク成長法により、ヘキサゴナール型単結晶よりなる種結晶上にヘキサゴナール型単結晶を成長させる半導体単結晶の製造方法において、前記種結晶の成長主面に接する側面は(0001)面及び(1-100)面のいずれの面からも傾いた面のみを有する種結晶を用いることを特徴とするものである。

【0008】

【作用】 本発明者らの研究の結果、成長面の凹面化の原因としてもっとも重要な点は、ヘキサゴナール型結晶である例えば α -SiCでは他の結晶に比べ結晶成長速度の面方位依存性が強く、ファセットと呼ばれる平面が出現することを見いだした。図11に(0001)面を成長主面(1-100)面をその側面にした場合のファセット111を示す。本発明者らは、この現象は<0001>、または<1-100>方位の成長速度が非常に遅いため、成長主面の側面の(0001)面或いは(1-100)面に平行に延びようとしその結果ファセットが表れると考えた。このファセット111の出現する部分は成長主面よりも成長速度が早く、成長する単結晶は中央部よりもその周辺部が盛り上がってくる（112は周辺部の盛り上がり）。その結果得られる単結晶の成長主面は、中央部がくぼんだ凹型になることを見いだした。

【0009】上記ファセットの出現を防止するには、発明者らは、種結晶の成長主面に接する側面を(0001)面及び(1-100)面のいずれの面からも傾けることにより達成できることを見いだした。

【0010】

【実施例】 以下に本発明の実施例を詳細に説明する。

【0011】本発明の第1の実施例を説明する。図1はバルク成長法の1つである昇華法単結晶製造装置の概略図である。11は種結晶、12はポーラスグラファイト、13は坩堝、14は坩堝蓋、15は原料粉末である。本実施例ではヘキサゴナール型SiC単結晶を成長

させた。

【0012】図2にヘキサゴナル型SiC種結晶11の結晶型と成長主面の関係を示す。また図3にSiC種結晶11の形状を表す図を示す。種結晶11の成長主面は{000-1}面((0001)の炭素面)とし、直径10mmの円形に整形した。また、側面はすべて{000-1}面に対し、45°になる様、頭の潰れた円錐状に加工した。この種結晶11の広い面の方を成長面として図1の装置を用い種結晶11上に原料を昇華させ、SiC単結晶を成長させた。成長条件としては、種結晶の温度2300℃、原料温度2500℃、成長圧力50torrとした。成長時間20時間で長さ約20mmのSiC単結晶を成長した。

【0013】図4に本発明により成長させたSiC単結晶41と比較例として成長主面を{000-1}面((0001)の炭素面)とし側面の一部に(1-100)面がでている円柱状の種結晶42を用い第1の実施例と同じ条件で成長させたSiC単結晶43の断面図を示す。

【0014】比較例(図4(b))の場合成長させたSiC単結晶43は樽型になり、やがて径が広がるのが止まってしまった。また、成長面は中央部で約3mmくぼんだ凹型となり、円周部の異常成長による応力により転移、双晶等の結晶異常が起こってしまった。これに対し、本発明(図4(a))ではSiC単結晶41は円錐状に成長する。このため、本発明の方が結晶径が大きくなり、大口径基板の製造を可能にすることができる。また成長主面の凹面化はほぼなくなり、円周部の異常成長による欠陥の増加を抑えることが可能となり結晶の品質を向上することができた。

【0015】図5に本発明の種結晶として成長主面を(001)面とした場合の側面の角度を示す。側面を(1-100)面((0001)面に対して90°)に対して少なくとも3°以上傾けることが望ましい(図中Aの領域)。このような種結晶を用いることによりファセットの発生を抑えることができ成長主面の凹面化を防ぐことが可能となる。従って、大口径で結晶欠陥の少ないヘキサゴナル型単結晶を成長させることができる。次に本発明の第2の実施例を示す。本実施例では成長主面を(11-20)面としたSiC種結晶を用いてSiC単結晶を成長させた。使用する装置は図1に示すものを用い成長条件は第1の実施例と同様に行った。

【0016】図6に本実施例の種結晶の α -6HSiC結晶型と成長主面である(11-20)面の関係の図を示し、図7に成長主面を(11-20)面とした場合の側面の角度を示す。成長主面を(11-20)面とした場合側面に(1-100)面及び(0001)面が表れるのは主面から30°、90°、150°でありこれらの面からそれぞれ3°以上傾ければ良い(図中Aの領域)。

【0017】本実施例では、成長主面を直径10mmの円状に形成し、側面を成長主面から60°傾けた頭の潰

れた円錐状の種結晶を用い、面の広い方を成長主面として成長長さ20mmのSiC単結晶を成長させた。本実施例においても第1の実施例と同様に円錐状にSiC単結晶を成長させることができた。また、円周部のファセットは表れず、成長面の凹面化は起こらなかった。次に本発明の第3の実施例を示す。本実施例では成長主面を(1-100)面としたSiC種結晶を用いてSiC単結晶を成長させた。使用する装置は図1に示すものを用い成長条件は第1の実施例と同様に行った。

【0018】図8に本実施例の種結晶の α -6HSiC結晶型と成長主面である(1-100)面の関係の図を示し、図9に成長主面を(1-100)面とした場合の側面の角度を示す。成長主面を(1-100)面とした場合側面に(1-100)面及び(0001)面が表れるのは主面から60°、90°、120°でありこれらの面からそれぞれ3°以上傾ければ良い(図中Aの領域)。

【0019】本実施例では、成長主面を直径10mmの円状に形成し、側面を成長主面から45°傾けた頭の潰れた円錐状の種結晶を用い、面の広い方を成長主面として成長長さ20mmのSiC単結晶を成長させた。本実施例においても第1の実施例と同様に円錐状にSiC単結晶を成長させることができた。また、円周部のファセットは表れず、成長面の凹面化は起こらなかった。

【0020】次に、本発明の第4の実施例を示す。本実施例では上記第1、2、3の実施例で用いたSiC種結晶の主面を更に研磨し、各主面から5°ずらした面を成長主面とする種結晶を用いてSiC単結晶を成長させた。成長装置は図1のものを使用し、成長条件は第1の実施例と同じものとした。本実施例では成長主面と側面の成長速度がほとんど変わらないため、結晶の形は種結晶から自然な円錐形に広がり、良好な単結晶を得ることができる。

【0021】次に、本発明の第5の実施例を示す。本実施例ではバルク成長法の1つである溶液法を用いてSiの溶液からSiC種結晶上にSiC単結晶を析出し成長させた。図10は本実施例で用いた成長装置である。CまたはSiCよりなる坩堝101中にSi溶媒102を溜めて、溶媒中の最高温部を1800℃、種結晶103の温度を1700℃として、100H成長を行う。るつぼのCと溶媒のSiがSiC種結晶103上で析出し単結晶が成長する。この場合種結晶は第1の実施例と同様に、成長主面を(0001)面、側面を成長主面から45°傾けて頭の潰れた円錐状に加工した。この種結晶の面の広い方を成長主面とし上記成長条件でSiC種結晶103上に10mm前後のSiC単結晶を成長させた。本実施例においても側面に(1-100)面がでている種結晶を用いたものに比べ大幅に結晶の径の拡大を図ることができ、成長面の凹面化はみられなかった。

【0022】次に、本発明の第6の実施例を示す。本実施例では昇華法を用いてヘキサゴナル型窒化ガリウム

の単結晶の成長を行った。現在窒化ガリウムは種結晶としてあまり大きな結晶が得られないため、微小な結晶を種として使用する必要がある。本実施例では、成長主面を(0001)面とし、側面を成長主面から 45° 傾いた頭のつぶれた円錐状に加工した種結晶を用いた。この側面はいずれも(1-100)面からは傾いた面となっている。成長装置は実施例1で使用了ものとはほぼ同じもので成長温度は原料は 1300°C 、種結晶 1200°C で N_2 雰囲気、圧力数Torrの条件で行った。本実施例においても大幅に成長単結晶の径の増大が図られ、成長面の凹面化はみられず品質の向上が図られた。

【0023】尚、上述した各実施例において成長主面は円形の種結晶を用いたが三角、四角、多角形、楕円形等特に限定するものではない。三角、四角、多角形の場合種結晶を柱状にしても成長主面に接する側面を(0001)面及び(1-100)面のいずれの面からも傾いた面のみにすることができる。また、上記した各実施例において結晶の温度、温度勾配は自由に変更して使用することができる。

【0024】

【発明の効果】上述したように本発明を用いることにより、結晶面の成長面が凹面になることを防ぎ結晶欠陥が少ない大口径のヘキサゴナル型半導体単結晶基板を製造することができる。またファセットがぶつかるところでの異常成長による欠陥の増加を抑えることができ結晶の品質を向上することができる。

【図面の簡単な説明】

【図1】 本発明の第1、2、3、4、6の実施例で用

いた昇華法単結晶製造装置の概略図

【図2】 ヘキサゴナル型結晶の(0001)面を表す図

【図3】 本発明の第1の実施例に用いた種結晶の形状を表す図

【図4】 本発明の第1の実施例で成長させたSiC単結晶と従来例との比較図

【図5】 成長主面を(0001)面としたときの種結晶の側面の角度を表す図

【図6】 ヘキサゴナル型結晶の(11-20)面を表す図

【図7】 成長主面を(11-20)面としたときの種結晶の側面の角度を表す図

【図8】 ヘキサゴナル型結晶の(1-100)面を表す図

【図9】 成長主面を(1-100)面としたときの種結晶の側面の角度を表す図

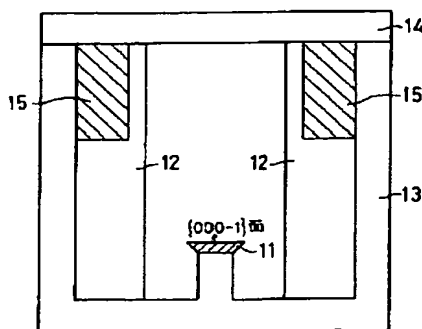
【図10】 本発明の第5の実施例で用いた溶液法単結晶製造装置の概略図

【図11】 成長主面を(0001)面その側面を(1-100)面としたときの円周部に出現するファセットを表す図

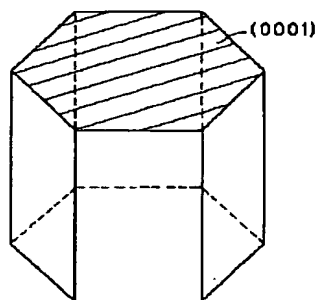
【符号の説明】

- 11. 種結晶
- 12. ポーラスグラファイト
- 13. 坩堝
- 14. 坩堝蓋
- 15. 原料粉末
- 101. グラファイト坩堝
- 102. Si溶媒
- 103. 種結晶

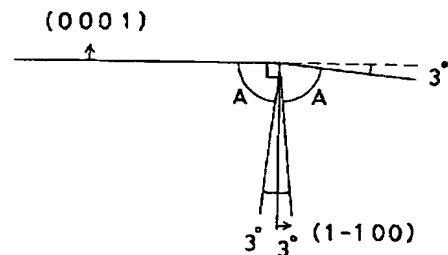
【図1】



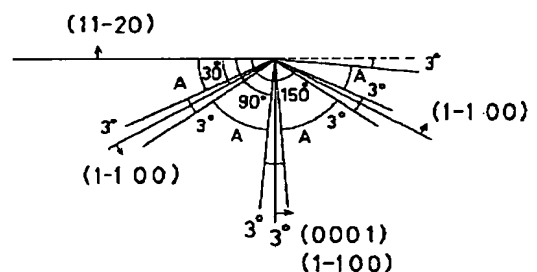
【図2】



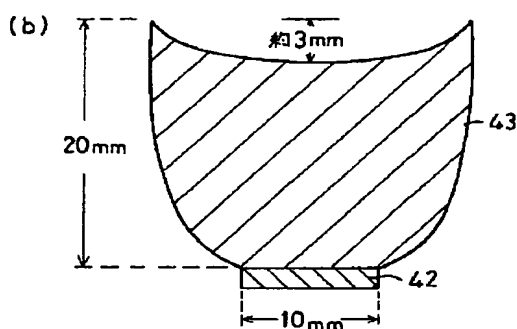
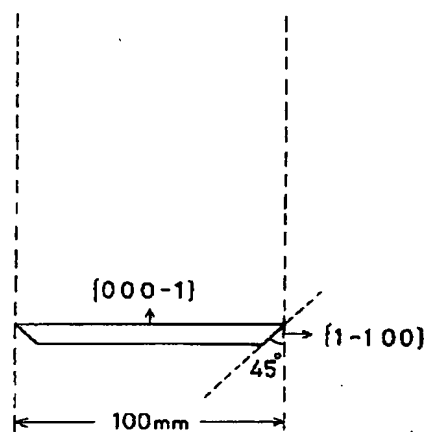
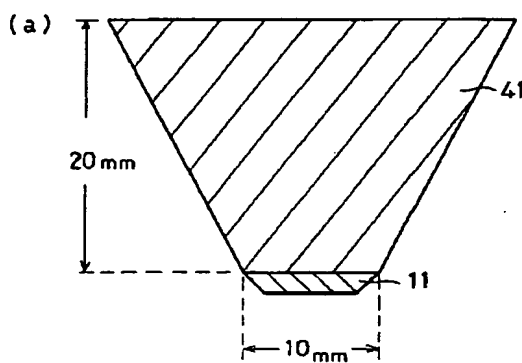
【図5】



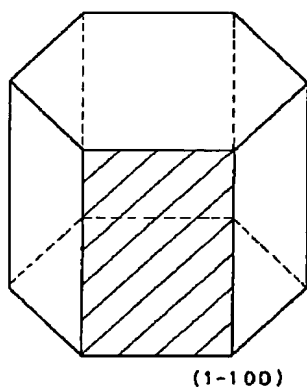
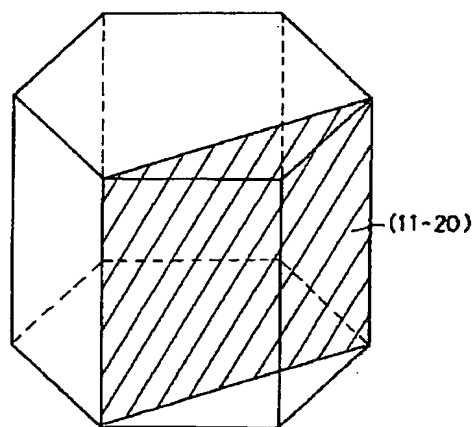
【図7】



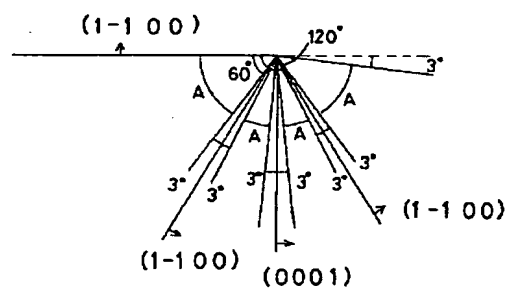
【図 4】



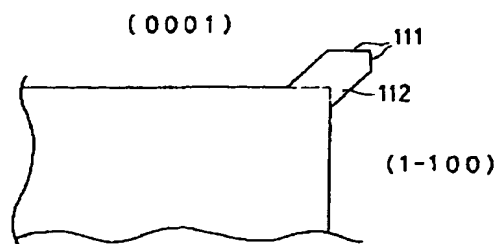
【图8】



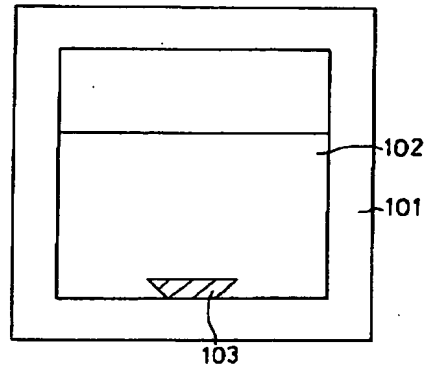
【図9】



【图 1-1】



【図10】



フロントページの続き

(51) Int. Cl. 5

C 3 0 B 29/36

// H 0 1 L 21/203

21/208

識別記号

庁内整理番号

F I

技術表示箇所

A 8216-4G

Z 8122-4M

D 9277-4M